

# The Deodorizing and Decolorizing of Degras

by Emily E. Grignard

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EMILE E. GRIGNARD

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## PREFACE

The investigations, which were made in order to prepare this article, were suggested to me by my brother Charles E. Grignard of the Sonneborn Sons Oil Co., and were carried out under the supervision of Dr. E.H.S.Bailey with much assistance and oversight by Dr. F.W.Bushong. I therefore take this opportunity to acknowledge my indebtedness to them for their aid and valuable suggestions during the time the work was being prepared.

It has been my purpose in this article to present a method for the deodorizing and decolorizing of Degras, or Wool-fat, that would make it commercially valuable.

*Emile E. Grignard.*

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## THE DEODORIZING AND DECOLORIZING OF DEGRAS

The foul and ill-smelling wash-waters from the washing of the wool fibers, have in the past been run direct into the streams and formed one of the most troublesome sources of pollution. They being difficultly saponifiable but readily emulsifiable, deposited themselves over the entire length of the stream. During the past few years the boards of health over various parts of the country have required that all substances, such as waste oils and fats be kept out of the brooks and rivers. It is only natural then that these waste products have assumed more importance. Among these products are the Distilled Grease Oleines, Sod Oil, Cotton-seed Foots, Fuller's Grease, Black Oil, Garbage Grease, and Wool Fat. This Wool Fat commonly goes under the names of British, German, or American Degras, Suint, Lanoline, Wool Grease, Recovered Grease, or Yorkshire Grease. The trade name for it however, is "Degras".

Degras or Wool Fat as the name denotes is the greasy material obtained in the washing of wool.

This greasy substance obtained from the sheep's wool, contains a large amount of fatty matter of a very peculiar character. It is excreted by all parts of the animal, but is found most abundantly about the breast and shoulders. Besides this greasy excretion, Degras is also contaminated with a number of other impurities. The composition of Degras may be classified as follows:

(a) Wool Grease -which occurs in large quantities as an external coating on the fiber, being the natural exudation of the sheep and serves as a protection to the fiber, preventing it from becoming felted and mechanically injured. It differs from other animal fats in that it does not consist of the glycerides of the fatty acids, and is very difficultly saponifiable with caustic alkalies. Wool Grease possesses more the chemical properties of a wax, as it is composed mostly of the higher solid alcohols known as cholesterin and isocholesterin both in the free state and as esters with the fatty acids. Though insoluble in water and not saponifiable by alkalies, cholesterin is easily emulsified, a property on which is based

the usual method of wool scouring. Wool Grease however, is easily soluble in naphtha and other volatile solvents. This Wool Grease amounts to 6 - 17 percent of the wool.

(b) Suint - or dried up perspiration, consisting largely of potash salts of organic acids such as oleic, valeric, and acetic acids, along with sulphates, phosphates, chlorides, and nitrogenous compounds, amounting to 5 - 24 percent of the wool. These are soluble in water.

(c) Miscellaneous Dirt - such as dust, sand, vegetable matters, manure, tar, etc. These amount to 19 - 39 percent of that of the wool.

The wool itself generally contains from 30 - 80 percent of impurities, consisting of the wool grease, suint, and dirt.

Unless freed from dirt, dust, burrs, etc., the wool cannot be spun or woven successfully, and it will not dye evenly if all the natural oil and grease is not removed. A scouring process is therefore necessary, as it removes all these substances quickly, cheaply, and without injury to the wool.

Scouring includes a number of operations,

of which the first is called dusting. This consisted in the early days of the industry of beating, shaking, and otherwise of removing the dust and dirt from the wool by hand. It is essentially a mechanical process, without the use of chemicals, and is now accompanied by machines known as dusters. These dusters operate on the principle of beating the wool over a screen, through which falls the dirt. The wool, having the dirt partly removed, is now scoured in a warm soap solution, to which more or less soda ash is added. The wool grease is easily emulsified by the alkaline soap solution, whereas the suint is dissolved by the water, and the other impurities are removed by the mechanical action of the water in the tanks.

The temperature of this scouring bath is important. While high temperatures facilitate scouring, the saving in time is effected at the cost of the luster and strength of the fibers. The temperature should range from 100 - 120 degrees F. It should never be over 120 degrees F. unless absolutely necessary in scouring very dirty stock, and then only for a short time. This scouring is

usually conducted in long tanks arranged in tandem order, and the fiber agitated by machinery in the least possible degree so as to avoid felting. The greasy wool is introduced into the first tank by means of a traveling apron, and is slowly carried through the scouring liquor by moving forks until it is caught up by squeeze rolls and passed into the second tank, where it is carried forward in the same manner through another soap solution; it is finally carried through a third tank containing fresh warm water for rinsing. The scouring liquor is introduced fresh into the second tank and passes thence into the first tank, so as to have the clean wool coming from the fresh liquor. To recover the grease, the wash waters are allowed to stand so as to settle out the sand and dirt and then these spent liquors are "soured" with sulphuric acid, in order to decompose the soaps and liberate the fatty matters. These, together with the associated wool grease, settle out on the surface of the liquor from which they are removed. The excess of water is eliminated by hot pressing in bags. This crude product so obtained is called

"Degras". The grease thus obtained, however, contains besides wool grease, the fatty acids of the soaps used and also traces of sulphuric acid. This Degras is of inferior quality to that obtained by the "solvent process" that is now so much in use in this country.

The greasy wool is treated with solvent naphtha, generally petroleum-naphtha, in closed kiers, and the resulting solution of wool grease is transferred to stills where the naphtha is recovered and the wool grease is obtained as a by-product. The degreased wool is next treated with a dilute warm soap solution to remove the suint and dirt. This process leaves the fiber in a much better condition and the recovered grease is of sufficient value to pay for the cost of scouring. The product thus obtained is of a lighter color and better quality than that obtained in the acid process. It is free from sulphuric acid, and practically so from fatty acids, and is the one to which the term wool grease or "Degras" is properly applied.

There is another form of waste fat on the

market called "Degras" that is obtained in the chamoising process, and largely used in dressing leather. It is rank stuff, low in cost, and only used in the leather industry.

The chamoising process consists essentially in oiling the suitably prepared skins with whale or cod oil, stamping them in the stocks and placing them in heaps, so that a fermentative change attended with a development of heat is brought about. The process is complete when the skins have acquired the usual yellow color of chamois leather. Under the considerations, oxidation of the oil takes place and a portion of it combines with the skin, from which it cannot be removed with the usual solvent. About an equal quantity of uncombined oil is also mechanically enclosed in the skin. After being well scraped with a blunt knife, by which much of the excess oil is removed, the skins are washed with lye and the emulsion treated with acid. The fatty matter which rises to the surface is added to the oil already obtained by scraping. The product so obtained constitutes a so-called degreas. But it is condemned as a lubricant for wool fiber.

The "Degras" with which I experimented with is known as Domestic Degras, and was purchased from the oil firm of Marden, Orth, & Hastings, of Chicago, Ill. This "Degras" is a dark brown substance, of a peculiar unpleasant odor, much like that of a stable and packing house combined. It is of a salvy consistence and has been used with lard, red, and mineral oils in the preparation of "compounded wool oils". These oils run from 25 - 75 percent cheaper than the pure lard, red, or olive oils. Such compounded wool oils have not been able to command much of a market price due to the odor which the Degras gives them. This smell is retained on the fabrics and consequently cannot be used in the lubrication of high class work. It is with this in mind that I endeavored to deodorize and decolorize this Degras, keeping constantly in view the minimum cost for the operation.

The composition of Degras is very complex, being a mixture of alcohols and esters. It is not a fat, but a collection of waxes. The esters present consist chiefly of those of cholesterol and its isomers. Some of the compounds that have been found in the grease are:



lanoceric acid	$C_{30}H_{60}O_4$
lanopalmic acid	$C_{16}H_{32}O_3$
myristic acid	$C_{17}H_{27}.CO_2H$
carnaubic acid	$C_{24}H_{48}O_2$

and other oily and volatile acids. Also:

carnaubyl	$C_{24}H_{48}O_2$
ceryl alcohol	$C_{27}H_{55}.OH$
ethyl alcohol	$C_2H_5.OH$
cholesterol	$C_{26}H_{43}.OH$

and isocholesterol have been found. It is almost impossible to give constants from such an uncertain mixture. The percentage composition of Degras according to Gill, Oil Analysis, 1911, Page 143, is:

Water	1%
Fatty Acids	19 - 26%
Neutral Oil	68 - 17%
Unsaponifiable	12 - 56%

From my experiments it seems almost impossible to get definite constants. The average of those I found are tabulated below:

Specific Grav.	0.901
Iodine Value	29.6
Saponification	98.8
Melting Point	41 C. degrees
Solidifying Point	30 C. degrees

The constants found by Sadtler, Industrial Organic Chemistry, Appendix Table, are:

Specific Grav.	0.901
Iodine Value	25 - 28
Saponification	98.2 - 102.4
Melting Point	39 - 42
Solidifying Point	30 - 30.2

DEODORIZING -

Degras mixes with water readily and forms emulsions that are unusually permanent. It absorbs as much as 80 percent water. After various experiments it was found best to remove the odor by the passage of steam through the grease at a certain definite temperature. Super-heated steam was tried but it could not be used as the temperature was too high breaking down some of the organic compounds and causing the brown color to turn to almost black and changing the other properties greatly. Passing ordinary steam through likewise did not succeed as the steam condensed upon entering the grease and caused an emulsion to be formed. As I stated above this emulsion is unusually permanent and if once formed it is almost high impossible to drive off the water with any simple method. But if the Degras is kept at a

temperature of 110 C. degrees by adjusting a flame under the grease containing vessel, the steam passes on through the Degras without condensing. And the obnoxious odors are carried right along with the steam. The amount of the smell passing through can be determined by condensing the steam and judging from the odor of the condensed steam or water. This condensation when it first comes over is almost indescribable with regard to its unpleasantness and rankness of smell. As the length of time increases the steam passing through gradually diminishes this smell until at the end of five to six hours it has practically disappeared. The testing of this disappearance in the odor is best accomplished by condensing some of the steam passing through and smelling of the condensed water. When this water gives no trace of the odor of the Degras in the original state it is considered free from the obnoxious odors. Upon cooling it is impossible to get a trace of the former smell. The physical properties remain about the same and there seems to be no change in the chemical structure.

DECOLORIZING -

The color of Degras is not easily changed from the dark brown to that of a lighter hue. Its structure is so complex that it cannot be treated as a fat, oil, or grease. It is a collection of waxes. Animal Charcoal or Fuller's Earth have little effect on the color when used independently, but when used in conjunction with each other, change the color considerably.

The "deodorized degreas" is placed in an open dish over a water-bath and kept in a liquid condition at the temperature of the bath and Animal Charcoal, that has been previously incinerated, is added and the whole mass agitated by means of a mechanical stirrer for half an hour. This mixes the grease and the charcoal completely, and allows each particle of the charcoal to come in contact with the grease. Fuller's Earth, previously incinerated, is then added to the grease and the charcoal mixture and the mass again agitated for an hour. This whole mass is kept at the temperature of the water-bath continuously. At the end of this time the whole mass is thoroughly mixed and it is

then allowed to stand for several days,

Just what is Fuller's Earth is a question often asked. It is a mineral consisting chiefly of a hydrous bisilicate of alumina. It occurs in beds, associated with chalk, oolite, etc., and is usually of a greenish-brown or a slate-blue color. Sometimes it is found white. It has an uneven earthy fracture and its appearance is dull. It is soft enough to yield readily to the nail, is greasy to the touch, scarcely adheres to the tongue, and falls to pieces in water with a hissing or puffing sound. It does not become plastic. In this country it is used chiefly for deodorizing, decolorizing, and clarifying of fats, oils, and greases. It has a remarkable cleansing and bleaching power on the fats and oils. It is very cheap costing but \$.005 per pound.

The analytical results on Fuller's Earth according to Mr. D.T.Day, J.I.E.C., Vol. 4, No. 12, Page 891, are as follows:

	Fuller's Earth (yellowish)	Fuller's Earth (bluish)
$\text{SiO}_2$	59.37	52.81
$\text{Al}_2\text{O}_3$	11.82	16.92

$\text{Fe}_2\text{O}_3$	6.27	3.78
$\text{CaO}$	6.17	7.40
$\text{MgO}$	2.09	2.27
$\text{Na}_2\text{O}, \text{K}_2\text{O}$	0.99	0.79
Loss on Ignition	13.19	14.29

Fuller's Earth is a high percentage of silica, and water in the sundried material. Hence the decolorizing agent mainly depends upon the high percentage of hydrates of silica. The earth absorbs the unsaturated hydrocarbons.

According to Messrs. Engler and Albrecht, J.I.E.C., Vol. 4, No. 12, Page 893, the action of Fuller's Earth has been ascribed to its physical properties alone. But it is the opinion now that the action of the earth depends both upon its physical and its chemical properties.

Animal Charcoal is not so unfamiliar. It is variously called Animal Charcoal, Bone Black, or Ivory Black. It is prepared from bones by heating them in closed retorts until they undergo the process of destructive distillation. Combustible gases and water, together with the vapors of the various salts of ammonia, and oil are given off.

Bone Black is left in the retort and it is reduced to coarse grains from about the size of small peas down to large pin-heads. It is extensively used in the arts for decolorizing liquids. The general mode of using this Animal Charcoal is to allow the colored liquid to percolate through a layer of the charcoal, when all color is arrested. The application of heat to the liquids before filtration greatly facilitates the decolorization, and where the volume of liquid to be operated upon is not great, the most expeditious method is to boil the liquid and bone-black together, and then strain through filter paper or cloth. The composition of bone-black is 10 percent pure charcoal, and 90 percent of earthy salts. The power of absorbing colors appears to be due to the porosity of the substance, and is not resident simply in the pure charcoal. Animal Charcoal likewise has great powers of absorbing odors, especially those of a disagreeable nature, such as those of animal matter passing into a state of putrefaction.

After letting the mass, composing of the grease, animal charcoal, and Fuller's Earth, stand

for several days, it is heated to the temperature of the water-bath. When entirely liquid it is passed thru a filter press, using several thicknesses of heavy cloth filter, or else it is allowed to filter directly. The resulting Degras that passes on through is of a light yellow-brown color and entirely devoid of the obnoxious smell of the crude material. If the quantity of the material to be filtered is of small bulk, it is best to use a solvent and pass the solution through a filter paper or cloth, and then distill over the solvent.

It is plainly seen that this process which is very successful is extremely low in cost.

Crude "Degras", according to statistics from the Bureau of Foreign and Domestic Commerce, Washington, D.C., is imported in the crude state at a cost of \$.019 per pound, and is sold wholesale by Marden, Orth, & Hastings, Chicago, Ill., for \$.03 $\frac{3}{4}$  per pound. Fuller's Earth costs \$.005 per pound, and Animal Charcoal costs \$.05 - \$.07 per pound in car load lots.

The amount of the decolorizers used is about one-fifth that of the volume of the Degras. These materials, however, can be used over and over

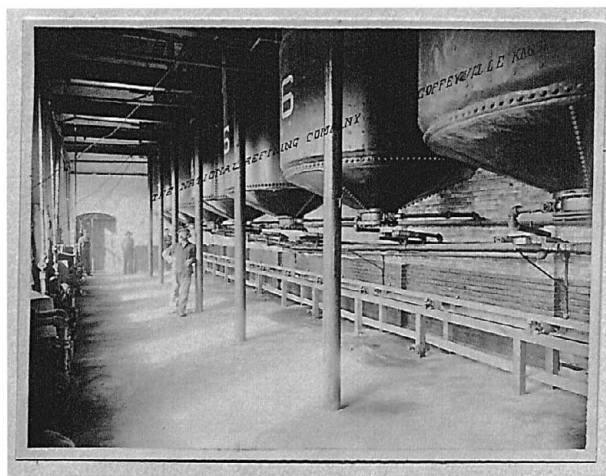


again. All that is necessary is simply to incinerate them again. By so doing the organic material that is absorbed is burned out and the decolorizers are again ready for use.

The necessary equipment for working this process on a commercial basis of 100,000 gallons per week, would consist of a plant containing the "Deodorizing Tanks", the "Decolorizing Tanks", the "Incinerating Furnaces", and the "Filter Presses".

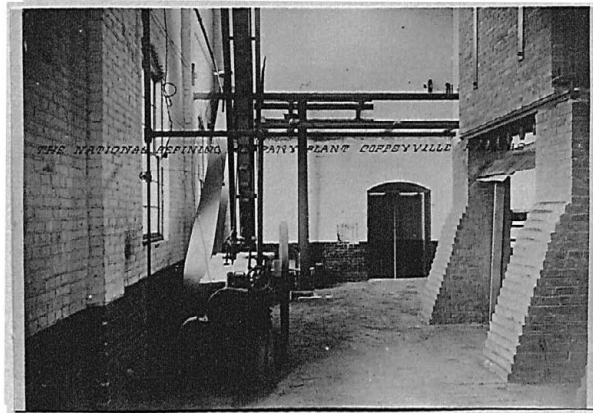
There should be about six "Decolorizing Tanks", 15 feet high with a diameter of 10 feet, cone-shaped at the top containing the steam exhaust. The steam enters at the bottom passing on through the grease to the top where it escapes. A tank of such dimensions would deodorize approximately 8500 gallons of the crude Degras every three days, or six such tanks, 102,000 gallons per week.

The "Decolorizing Tanks" like those used



at the National Refining Co., at Coffeyville, Kans. are 20 feet high and 10 feet in diameter. They contain approximately 11,500 gallons each. Of this volume 2,500 gallons should be devoted to the Fuller's Earth and the Animal Charcoal, and the remaining 9,000 gallons to the deodorized grease. To have a capacity of 100,000 gallons per week, it would take six such tanks running two shifts per week.

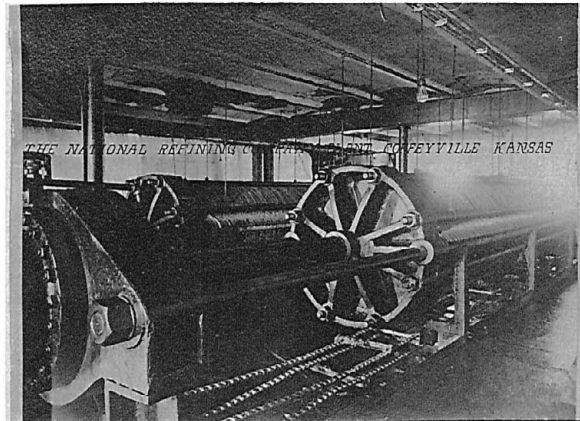
The "Incinerating Furnaces", two in num-



ber, would be amply sufficient, and they could be run in conjunction with the production of the steam. These furnaces would incinerate the earth and charcoal just previous to the addition of them at the change of shifts.

The filter presses on the market are large enough to take care of the deodorized and decolorized grease. Their capacity is known, and the number of them to

to be used is easily determined. These presses



should be steam jacketed to facilitate filtering.

The employ of three or four men should be sufficient to run a plant of this size. The National Refining Co. of Coffeyville, Kans. employ not more than four to look after the refining as the pictures easily show.

Once the plant is in running order, the approximate cost to refine 100,000 of the crude Degras, in gallons, would amount to:

Labor, \$60. per. (3)men,	45.00
Fuller's Earth, @ .005 per	
lb., 30,000 lbs.	150.00
Charcoal, @ .07 per lb.	
15,000 lbs.	1050.00
Steam, Fuel, etc.	100.00
	<hr/>
	\$ 1345.00

Since 100,000 gallons cost approximately \$1345.00

1 gallon costs -- \$0.01345

About 8 lbs. of Degras are contained in 1 gallon

Then to refine 1 lb. crude Degras costs \$0.00168

This increase in cost is decidedly low,  
being approximately one-fifth of one cent per pound.

That added to the cost of the crude material,

\$0.019

\$0.00168

\$0.02068

makes it but a little over two cents per pound.

This price is under that of Marden, Orth, & Hastings  
of Chicago, Ill. price for their crude, ill-smell-  
ing stuff, which they sell for \$0.0375 wholesale.

The compounded wool oils now used in the  
textile industry cost from 19 - 22 cents to manu-  
facture. Their composition varies, but it is  
generally composed of:

Degras	15%
Lard Oil	5%
Red Oil	5%
Mineral Oil	75%

Though it costs about twenty cents to prepare, it commands little market value due to its odor.

There are compounded wool oils without the use of Degras but they cost much more to prepare. An oil that does not contain the ill-smelling grease commands a market value of almost twice that that contains the Degras.

The advantage of using this odorless and pale colored Degras in the wool oil compounds is clearly seen from a financial standpoint.

Finis.

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